



Article

Comparison of Chemical Properties between Traditional and Commercial Vinegar

Sina Cosmulescu ^{1,*} , Ana-Maria Stoenescu ^{2,*} , Ion Trandafir ³ and Felicia Tuțulescu ¹¹ Department of Horticulture & Food Science, Horticulture Faculty, University of Craiova, A.I. Cuza Street 13, 200585 Craiova, Romania; felicia.tutulescu@edu.ucv.ro² Department of Biology & Environmental Engineering, Horticulture Faculty, University of Craiova, A.I. Cuza Street 13, 200585 Craiova, Romania³ Department of Chemistry, Science Faculty, University of Craiova, Calea Bucuresti Street 107, 200478 Craiova, Romania; ion.trandafir@edu.ucv.ro

* Correspondence: sina.cosmulescu@edu.ucv.ro (S.C.); anamaria.stoenescu@edu.ucv.ro (A.-M.S.)

Abstract: Fruits of wild fruit species are considered healthy foods with the potential to treat and prevent chronic diseases. In recent years, the food industry and consumers have become increasingly interested in the nutritional value and safety of food and ingredients. A traditional method of processing fruits from spontaneous flora is in the form of vinegar by simple and easy techniques. The aim of this paper was to analyze the chemical composition of homemade vinegars obtained by traditional methods from the fruits of some wild fruit species, compared with commercial vinegars. To evaluate the characteristics of the two types of vinegar, analyses were performed regarding the physicochemical properties (density, soluble dry matter, total titratable acidity, and pH) and the content of phenolic compounds using HPLC. In terms of pH, it varied between 2.58 and 3.67 for homemade vinegar and between 2.22 and 2.86 for commercial vinegar. The density of vinegar varied between 1015 and 1070 kg/m³ in the case of homemade vinegar and between 1013 and 1030 kg/m³ in the case of commercial vinegar. The phenolic compounds, gallic acid, neochlorogenic acid, catechin hydrate, chlorogenic acid, vanillic acid, caffeic acid, syringic acid, epicatechin, *p*-coumaric acid, ferulic acid, sinapic acid, salicylic acid, ellagic acid, rutin, and myricetin were found in homemade vinegar. Fewer phenolic compounds were identified in commercial vinegars. The data obtained highlight the high quality of homemade traditional vinegars compared with commercial ones. The biochemical composition of vinegars traditionally obtained from wild fruits, through simple recipes, demonstrates their role and importance for human well-being and the potentially beneficial effects on health.

Keywords: composition; HPLC; phenolic compounds; wild fruits

Citation: Cosmulescu, S.; Stoenescu, A.-M.; Trandafir, I.; Tuțulescu, F. Comparison of Chemical Properties between Traditional and Commercial Vinegar. *Horticulturae* **2022**, *8*, 225. <https://doi.org/10.3390/horticulturae8030225>

Academic Editor: Massimo Lucarini

Received: 1 February 2022

Accepted: 3 March 2022

Published: 5 March 2022

Publisher's Note: MDPI stays neutral with regard to jurisdictional claims in published maps and institutional affiliations.



Copyright: © 2022 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (<https://creativecommons.org/licenses/by/4.0/>).

1. Introduction

Wild fruit species are considered rich in bioactive compounds [1–3] such as anthocyanins and flavonoids, and fruits are considered healthy food with potential in the treatment and prevention of chronic diseases [4]. Wild fruits are part of provisioning services (ecosystem goods), a component of forest ecosystem services [5]. In recent years, the food industry and consumers have become increasingly interested not only in the nutritional value and safety of food and ingredients, but also in being natural, organic, or healthy [6,7]. Interest in consuming natural antioxidants has increased considerably thanks to their antiviral, anti-inflammatory, and antihypertensive properties, and daily intake of phenol-rich foods could prevent chronic, degenerative, and coronary diseases such as cancer and atherosclerosis [8,9]. A product frequently used in both food and therapeutic medicine is vinegar because of its antimicrobial properties, combating obesity, diabetes, cardiovascular diseases, and cancer [10]. Vinegar is a natural food product resulting from carbohydrate-rich products, as a result of an alcoholic fermentation process followed by an acetic fermentation [10]. According to Budak et al. [11], traditional vinegars are produced

from raw materials (grapes, apples, plums, coconut, rice, potatoes, and tomatoes) that contain sugar or starch and require a two-stage fermentation [12] with initial production of ethanol and subsequent production of acetic acid. The same authors mention acetic acid as having a strong antimicrobial and antifungal activity. One way of using vinegar is in the form of disinfectant owing to its low pH, and many researchers confirm its beneficial effect on *Candida albicans* [13,14], *Escherichia coli* [11,14], *Klebsiella pneumoniae* [15], and *Staphylococcus aureus* [14]. Moreover, Baldas et al. [16] confirms the use of apple vinegar and grape vinegar as a disinfectant for fruits and vegetables in Turkey. Organic acids (acetic, propionic, malic, tartaric, and citric acids) are considered to be very important in the aroma of vinegars and are mainly produced during the fermentation process [17]. Phenolic compounds present in vinegars come from the raw material used, but changes may occur due to different processing methods and the plant material. Traditional vinegar processing methods are considered to have an influence on taste and aroma owing to the slower fermentation process [18]. The purpose of this paper was to analyze the physicochemical composition as well as the content of phenolic compounds of vinegars obtained by traditional methods from wild fruits, and to perform comparative analysis with commercial vinegars.

2. Materials and Methods

2.1. Material

Fruits from five wild fruit species from Bratovoesti forest area (44°05′29.3″ N 23°54′00.2″ E), Dolj County, Oltenia region, Romania were harvested by hand from several individuals of the same species for a high degree of homogeneity: *Crataegus monogyna* (L.) Jacq.—red hawthorn, *Crataegus pentagyna* (L.) Waldst. et Kit.—black hawthorn, *Pyrus pyrausta* (L.) Burgsd.—European wild pear, *Malus sylvestris* (L.) Mill.—European crab apple, and *Rosa canina* (L.)—rosehips, during the phenophase BBCH 87, fruit ripe for picking (principal growth stage 8). A total of 10 kg of fresh fruits from each fruit species collected over the suitable harvesting time in the year 2020 was used for vinegar preparation. For comparison, several types of vinegar were purchased from the supermarket, as follows: apple vinegar (5% acidity); unfiltered BIO organic apple cider vinegar (5% acidity) with a nutritional value of 15 kcal/100 mL, 0 lipids, carbohydrates, and proteins; Greek apple cider vinegar (6% acidity); unfiltered, raw, and unpasteurized BIO organic rosehip vinegar (4.5% acidity) with a nutritional value/100 mL of 19 kcal, lipids <0.1, carbohydrates 0.2 g, proteins <0.1 g; white wine (9% acidity); red wine (6% acidity); Greek grapes (6% acidity) with a nutritional value/100 mL of 0 g of proteins and lipids, 0.9 g of carbohydrates, and 0.3 g of sugars; quince (5% acidity); and sour cherry (7% acidity) with a nutritional value of 14 kcal/100 mL, 0.15 g of lipids, 2.55 g of carbohydrates, 0.10 g of fiber, and 0.72 g of proteins (all data have been retrieved from the product label). Three commercial vinegar bottles from each batch were used for laboratory determinations.

2.2. Methods

2.2.1. Vinegar Preparation

Homemade vinegars were prepared according to a traditional recipe as follows: 1 L of water (network water that meets all drinking parameters) and 50 g of beet sugar (Bod Sugar Factory, Bod, Brasov County, Romania) were added to 1 kg of previously chopped, cleaned, and washed fruits. The obtained mixture was stored in glass vessels and covered with cloth for 14 days at room temperature (22–25 °C) with daily stirring. After that, the mixture was filtered several times using filter paper (3–5 µm pore) until a liquid without impurities was obtained. The fermentation process was conducted at room temperature (22–25 °C) for a time period of 60 days (without stirring) in the absence of starter cultures. No selected cultures were added. Fermentation was performed by microorganisms present in the natural microflora of fruits.

2.2.2. Physicochemical analysis

The density (kg/m^3) was determined using a densimeter; the soluble dry matter content was determined using a HANNA Instruments HI96801 sucrose refractometer (0–85% Brix), Woonsocket, USA, with a precision of $\pm 0.2\%$ [19]; the total titratable acidity was determined by titrating the vinegars with 0.1 N NaOH solution using phenolphthalein as indicator [20] and the results were expressed in g acetic acid; the pH was determined at a temperature of 25 °C using a pH meter HANNA Instruments HI 255.

2.2.3. HPLC Analysis

The HPLC analysis was performed on an ultra-high performance liquid chromatograph (UltiMate 3000 XRS Liquid Chromatograph, Thermo Scientific, Waltham, MA, USA) combined with a Dionex UltiMate 3000 XRS Autosampler, an XRS pump, and an RS Diode UV–VIS matrix detector. The compounds were separated by reversed phase chromatography and were detected by absorbance and quantified with external calibration graphs. For the procedure, 1 mL of filtered vinegar (using a 0.45 μm PTFE microfilter) was used. To determine the phenolic compounds, vinegar samples were analysed using the method described by Stoenescu et al. [21] as follows: for the detection of compounds, a Thermo Scientific (Lithuania), Hypersil GoldTM (150 $\times$ 4.6 mm, 5 μ particle size) column was used, at a maintained temperature of 25 °C, a flow rate of 0.8 mL/min, and an injection volume of 5 μL . The order of appearance on the chromatogram of phenolic compounds was as follows: gallic acid, neochlorogenic acid, (+)-catechin hydrate, chlorogenic acid, vanillic acid, caffeic acid, syringic acid, (–)-epicatechin, *p*-coumaric acid, ferulic acid, sinapic acid, salicylic acid, ellagic acid, rutin, and myricetin. The mobile phase consisted of 1% aqueous acetic acid solution (B) and 100% methanol (C). The samples were eluted with the following gradient: 90% B and 10% C from 0 to 6 min, 84% B and 16% C from 7 to 25 min, 72% B and 28% C from 26 to 37 min, 65% B and 35% C from 38 to 47 min, 50% B and 50% C from 48 to 64 min, and 90% B and 10% C from 65 to 70 min, in order to restore the initial conditions, before injection of a new sample. The detection of phenolic compounds was performed by UV absorption at $\lambda = 278$ nm. All standards and reagents were purchased from Sigma-Aldrich. All vinegar determinations were made at 3 months of age. Integration, data storage, and processing were performed by Thermo ScientificTM DionexTM ChromeleonTM CDS software.

2.2.4. Statistical Analysis

All results were expressed in mg L^{-1} of vinegar and represent the mean and standard deviation of three consecutive determinations processed in XLSTAT, Addinsoft. Statistical analysis was performed with IBM SPSS Statistics 26 software. One-way ANOVA and Duncan multiple range tests at $p < 0.05$ were used.

3. Results

In order to highlight the nutritional value of traditional vinegars, determinations were made regarding the physicochemical characteristics (density, soluble dry matter, total titratable acidity, and pH) [22,23] and the content of phenolic compounds. Following the statistical processing of the obtained data (one-way ANOVA), there were significant differences found between homemade vinegars and commercial vinegars ($p < 0.05$), highlighted by the results of the Duncan multiple range tests (Tables 1–3).

The results regarding the physicochemical properties are presented in Table 1. In terms of pH of homemade vinegars, the lowest value was identified at *Malus sylvestris* vinegar (2.58) and the highest at *Crataegus monogyna* vinegar (3.67), compared with commercial vinegars, which had values between 2.22 (quince vinegar) and 2.86 (apple vinegar). In terms of vinegar density, this varied in the case of homemade wild fruits vinegar between 1015 kg/m^3 for *Pyrus pyraeaster* vinegar and 1070 kg/m^3 for *Crataegus pentagyna* vinegar, and in the case of commercial vinegars between 1013 kg/m^3 for BIO rosehip vinegar and 1030 kg/m^3 for sour cherry vinegar. The soluble dry matter content (Brix%) varied in the case of traditionally obtained vinegars between 3.2% corresponding to *P. pyraeaster* vinegar

and 15.6% corresponding to *C. pentagyna* vinegar. In the case of commercial vinegars, the soluble dry matter was situated between 3.6% for Greek grapes vinegar and 8.1% for sour cherry vinegar.

The total titratable acidity of homemade vinegars was 0.32 g acetic acid/100 mL for *P. pyraeaster* vinegar and a maximum of 5.09 g acetic acid/100 mL for *M. sylvestris* vinegar. In comparison, commercial vinegars had values between 4.14 (BIO rosehip vinegar) and 9.63 (sour cherry vinegar) g acetic acid/100 mL.

Table 1. Physicochemical properties of the homemade and commercial vinegars.

Vinegar Type	Vinegar	pH	Vinegar Density (kg/m ³)	Brix (%)	Total Titratable Acidity (g Acetic Acid/100 mL)
Homemade	European crab apple	2.58 ± 0.10 ^{f,g,*}	1040 ± 0.66 ^b	9.5 ± 0.05 ^b	5.09 ± 0.12 ^d
	European wild pear	3.56 ± 0.01 ^{a,b}	1015 ± 0.30 ^{l,j}	3.2 ± 0.03 ^j	0.32 ± 0.01 ^k
	Rosehip	3.42 ± 0.01 ^b	1027 ± 0.42 ^d	7.3 ± 0.02 ^d	1.04 ± 0.03 ^j
	Red hawthorn	3.67 ± 0.01 ^a	1020 ± 0.31 ^f	9.3 ± 0.03 ^b	1.58 ± 0.05 ⁱ
	Black hawthorn	3.25 ± 0.01 ^c	1070 ± 0.25 ^a	15.6 ± 0.03 ^a	1.92 ± 0.12 ^h
	Sour Cherry	2.32 ± 0.16 ^{h,i}	1030 ± 0.32 ^c	8.1 ± 0.09 ^c	9.63 ± 0.21 ^a
Commercial	Apples	2.86 ± 0.16 ^d	1015 ± 0.35 ^{i,j}	3.9 ± 0.03 ^h	5.07 ± 0.19 ^d
	White wine	2.48 ± 0.01 ^{g,h}	1024 ± 0.46 ^e	4.4 ± 0.14 ^f	9.32 ± 0.09 ^b
	Red wine	2.44 ± 0.01 ^{g,h}	1015 ± 0.40 ^j	4.0 ± 0.18 ^{g,h}	4.95 ± 0.04 ^{d,e}
	Quince	2.22 ± 0.07 ⁱ	1016 ± 0.25 ^{h,i}	3.9 ± 0.08 ^h	4.77 ± 0.06 ^{e,f}
	Apples BIO	2.68 ± 0.01 ^{e,f}	1018 ± 0.38 ^g	5.1 ± 0.13 ^e	4.61 ± 0.06 ^f
	Rosehips BIO	2.76 ± 0.01 ^{d,e}	1013 ± 0.31 ^k	4.4 ± 0.09 ^f	4.14 ± 0.09 ^g
	Apples Greece	2.27 ± 0.01 ⁱ	1016 ± 0.40 ^{i,j}	4.2 ± 0.23 ^{f,g}	6.98 ± 0.05 ^c
	Grapes Greece	2.46 ± 0.01 ^{g,h}	1017 ± 0.45 ^{g,h}	3.6 ± 0.10 ⁱ	5.15 ± 0.07 ^d

Mean ± standard deviation ($n = 3$). * Different letters indicate statistically significant differences (Duncan's multiple range test, $p < 0.05$).

Table 2. Phenolic compounds' content (mg L⁻¹) of the homemade vinegars.

Phenolic Compounds	European Crab Apple	European Wild Pear	Rosehip	Black Hawthorn	Red Hawthorn
*GA	12.31 ± 0.26 ^{d,**}	10.88 ± 0.23 ^{d,e}	56.47 ± 1.20 ^b	126.40 ± 2.68 ^a	n.d.
NCHA	19.88 ± 0.42 ^d	9.37 ± 0.20 ^e	339.42 ± 7.20 ^b	353.90 ± 7.51 ^a	70.57 ± 1.50 ^c
C	25.36 ± 0.54 ^d	n.d.	674.83 ± 14.32 ^a	48.54 ± 1.03 ^c	286.52 ± 6.08 ^b
CHA	462.15 ± 9.80 ^a	53.41 ± 1.13 ^d	n.d.	66.60 ± 1.41 ^c	356.50 ± 7.56 ^b
VA	n.d.	0.89 ± 0.02 ^e	99.13 ± 2.10 ^a	31.83 ± 0.68 ^b	16.66 ± 0.35 ^c
CFA	10.91 ± 0.23 ^d	9.11 ± 0.19 ^d	123.53 ± 2.62 ^a	51.11 ± 1.08 ^b	15.72 ± 0.33 ^c
SYA	n.d.	n.d.	40.43 ± 0.86 ^a	0.97 ± 0.02 ^b	0.70 ± 0.01 ^b
EC	n.d.	21.32 ± 0.45 ^d	97.82 ± 2.08 ^b	62.02 ± 1.32 ^c	630.90 ± 13.38 ^a
pCA	n.d.	1.02 ± 0.02 ^e	262.35 ± 5.57 ^a	16.19 ± 0.34 ^d	41.82 ± 0.89 ^b
FA	n.d.	0.57 ± 0.01 ^d	2.65 ± 0.06 ^c	33.00 ± 0.70 ^a	31.77 ± 0.67 ^b
SA	n.d.	3.42 ± 0.07 ^d	50.73 ± 1.08 ^a	26.29 ± 0.56 ^c	36.73 ± 0.78 ^b
SAA	n.d.	3.37 ± 0.07 ^d	89.78 ± 1.90 ^b	88.20 ± 1.87 ^b	n.d.
EA	n.d.	1.04 ± 0.02 ^d	190.57 ± 4.04 ^a	1.73 ± 0.04 ^d	154.08 ± 3.27 ^b
RUT	n.d.	1.96 ± 0.04 ^b	n.d.	3.07 ± 0.07 ^a	n.d.
MYR	92.87 ± 1.97 ^a	2.33 ± 0.05 ^d	7.17 ± 0.15 ^b	4.66 ± 0.10 ^c	4.29 ± 0.09 ^c

*GA—gallic acid; NCHA—neochlorogenic acid; C-(+)—catechin hydrate; CHA—chlorogenic acid; VA—vanillic acid; CFA—caffeic acid; SYA—syringic acid; EC-(—)—epicatechin; pCA—*p*-coumaric acid; FA—ferulic acid; SA—sinapic acid; SAA—salicylic acid; EA—ellagic acid; RUT—rutin; MYR—myricetin. Mean ± standard deviation ($n = 3$); "n.d." not detected. ** Different letters indicate statistically significant differences (Duncan's multiple range test, $p < 0.05$).

Table 3. Phenolic compounds content (mg L^{−1}) of the commercial vinegars.

Phenolic Compound	Sour Cherry	Apples	White Wine	Red Wine	Quince	Apples BIO	Rosehips BIO	Apples Greece	Grapes Greece
*GA	n.d.	10.23 ± 0.22 ^e	n.d.	10.73 ± 0.23 ^{d,e}	0.42 ± 0.01 ^{g,h}	2.28 ± 0.05 ^g	0.25 ± 0.01 ^h	16.93 ± 0.36 ^c	8.11 ± 0.17 ^f
NCHA	1.06 ± 0.02 ^{f,**}	0.97 ± 0.02 ^f	0.22 ± 0.01 ^f	1.08 ± 0.02 ^f	3.61 ± 0.08 ^{e,f}	0.03 ± 0.01 ^f	3.08 ± 0.07 ^{ef}	n.d.	2.36 ± 0.05 ^{e,f}
C	n.d.	4.78 ± 0.10 ^f	3.08 ± 0.07 ^f	1.97 ± 0.04 ^f	n.d.	n.d.	n.d.	15.33 ± 0.33 ^e	8.10 ± 0.17 ^{e,f}
CHA	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0.99 ± 0.02 ^e	n.d.	n.d.
VA	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0.54 ± 0.01 ^e	2.51 ± 0.05 ^d	n.d.
CFA	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0.90 ± 0.02 ^e	n.d.
pCA	n.d.	n.d.	35.43 ± 0.75 ^c	n.d.	n.d.	n.d.	n.d.	0.35 ± 0.01 ^e	n.d.
SAA	n.d.	14.24 ± 0.30 ^c	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	100.44 ± 2.13 ^a
EA	n.d.	n.d.	69.02 ± 1.46 ^c	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
MYR	n.d.	n.d.	n.d.	n.d.	n.d.	2.51 ± 0.05 ^d	n.d.	n.d.	n.d.

*GA—gallic acid; NCHA—neochlorogenic acid; C(+)—catechin hydrate; CHA—chlorogenic acid; VA—vanillic acid; CFA—caffeic acid; pCA—*p*-coumaric acid; Acid; SAA—salicylic acid; EA—ellagic acid; MYR—myricetin. Mean ± standard deviation (*n* = 3); “n.d.” not detected. ** Different letters indicate statistically significant differences (Duncan’s multiple range test, *p* < 0.05).

The differences between the different types of vinegars and the different species indicate a high degree of variability in these analyzed parameters.

The results on the composition of phenolic compounds of traditional and commercial vinegars are presented in Tables 2 and 3. Phenolic compounds have many bioactive properties and food intake has beneficial effects on human health [24]. Predominant in the case of *M. sylvestris* vinegar was chlorogenic acid with 426.15 mg L^{−1} of vinegar, with the results in accordance with the literature.

The same vinegar had the highest content of myricetin (92.87 mg L^{−1}). *C. pentagyna* vinegar had the highest values for gallic acid (126.40 mg L^{−1}), followed by neochlorogenic acid (353.90 mg L^{−1}), ferulic acid (33.00 mg L^{−1}), and rutin (3.07 mg L^{−1}). *R. canina* vinegar had the highest values for catechin hydrate (674.83 mg L^{−1}), followed by *p*-coumaric acid (262.35 mg L^{−1}), ellagic acid (190.57 mg L^{−1}), caffeic acid (123.53 mg L^{−1}), vanillic acid (99.13 mg L^{−1}), sinapic acid (50.73 mg L^{−1}), and syringic acid (40.43 mg L^{−1}).

C. monogyna vinegar was noted for its highest epicatechin value (630.90 mg L^{−1}). Commercial white wine vinegar had the highest value of salicylic acid, at 100.44 mg L^{−1}. From the analysis of the data obtained, it is possible to notice the difference between the homemade vinegars traditionally produced from wild fruit species and the commercial vinegars that have gone through different processing stages, in laboratory conditions. The content of phenolic compounds in wild fruit vinegars was much higher compared with commercial vinegars.

4. Discussion

Vinegar quality depends on the raw material and the applied fermentation process [25]. According to the legislation [26], apple vinegar must meet the following conditions: total acidity—minimum 50 g/L, expressed in pure acetic acid; residual alcohol content—more than 0.5% v/v—volume fraction; organoleptic and physico-chemical characteristics must technically meet the specifications of the manufacturers; the eligibility of the contamination level must be in accordance with the regulations in force. Ousaid et al. [1] mention the different vinegar processing techniques as having an influence on the quality and offered health benefits. With reference to the pH of apple vinegar, the values in the literature are different: 3.18–3.83 [27], 2.71–3.56 [28,29], and 2.91–3.20 [30], values higher than those obtained in this paper, where the limits of variation ranged between 2.27 and 2.86. Grape vinegar had a lower pH value (2.46) compared with those found by Ozturk et al. [28] with a pH between 2.70 and 3.90 and Matloob [29] with values between 2.49 and 2.99. Pashazadeh et al. [31] mention a pH of rosehip vinegar of 3.70, a value close to that obtained in the present study for *R. canina* vinegar (3.42), while Kalemba-Drozd et al. [32] obtained a pH of 2.85 for the same species. Chochevska et al. [33] confirms a similar pH value of 3.4 at homemade rosehip vinegar. Özdemir et al. [25] identified a pH of hawthorn vinegar (*C. tanacetifolia*) of 3.63, similar to the value obtained for *C. monogyna* (3.67) and higher than *C. pentagyna* vinegar (3.25). Karadag et al. [34] mention, for vinegars produced according to traditional recipes, a pH of 2.54 for rosehip vinegar, 2.72 for apple vinegar,

and 2.76 for hawthorn vinegar. Regarding the soluble dry matter content of this study, it ranged from 3.2% (*P. pyraeaster*) to 15.6% (*C. pentagyna*) in homemade vinegars and from 3.6% (Greek grapes) to 8.1% (sour cherry) in commercial vinegars (Table 1). The values in the literature regarding this parameter are different. Ozturk et al. [28] mention a Brix (%) between 1.22 and 20.80 for grape vinegar, between 1.02 and 12.90 for apple vinegar, and a value of 1.26 for hawthorn vinegar. Özdemir et al. [25] mention a value of 5.45% for *C. tanacetifolia* vinegar. Compared with the traditional vinegars used in the present study, Karadag et al. [34] identified a value of 4.01% for rosehip vinegar, lower than that obtained for traditional vinegar (7.3%); 4.17% for apple vinegar, lower than that obtained for traditional vinegar (9.5%); and 3.17% corresponding to hawthorn vinegar, a much lower value than that obtained in homemade vinegars (9.3 and 15.6%). Pashazadeh et al. [31] mention a soluble substance content of the black rosehip vinegar (*Rosa pimpinellifolia*) of 5.65%, a lower value than that obtained in the case of traditional vinegar (7.3%), but higher than that of commercial BIO vinegar (4.4%). Sung et al. [30] identified values between 8.6 and 13.4% in the case of analyzed apple vinegar.

In terms of acidity, in this paper, traditional homemade vinegars had values between 0.32 and 5.09 g acetic acid/100 mL, compared with commercial vinegars that had higher values between 4.14 and 9.63 g acetic acid/100 mL. Total titratable acidity for grape vinegar analyzed by Ozturk et al. [28] had values between 0.32 and 5.72, similar to those obtained in the present study for traditional vinegars, and Matloob [29] mentions values between 4.63 and 6.18%. Budak and Guzel-Seydim [35] identified a total titratable acidity for traditionally obtained red wine vinegar with a value of 85.15 g/L and a value of 122.97 g/L in the case of industrial red wine vinegar. Sung et al. [30] mention a value of 12% for apple cider vinegar, and Beceanu and Anghel [36] mention a total titratable acidity of 5.39 g/100 mL acetic acid for apple vinegar. Chochevska et al. [33] identified a total acid content of 17.4 g/L in homemade rosehip vinegar, a higher value compared with the one obtained in this study. It is found that there is a difference for this parameter, depending on the method of production and the raw material used.

Regarding the phytochemicals content of vinegars, many researchers have evaluated the profile of present bioactive compounds [1,37,38] in both industrial and artisanal vinegars. In the present study, 15 phenolic compounds (flavonoids and phenolic acids) were analyzed (Tables 2 and 3). Regarding the presence of phenolic compounds in apple vinegar, the results in the literature highlight a great variability. Thus, Kelebek et al. [39] identified gallic acid (0.47–2.57 mg L⁻¹), catechin (0.14–0.95 mg L⁻¹), chlorogenic acid (2.96–16.29 mg L⁻¹), and caffeic acid (0.19–1.77 mg L⁻¹); Nakamura et al. [40] mentioned chlorogenic acid (3.1–19.6 mg/100 mL) and caffeic acid (0–0.76 mg/100 mL); Bortolini et al. [41] confirmed the presence of caffeic acid (0.51–3.87 mg L⁻¹); and Liu et al. [42] mentioned chlorogenic acid with variation limits between 0.11 and 10.91 µg/mL. Compared with the results found in the literature, in this study, European crab apple vinegar (*M. sylvestris*) had a higher content of phenolic compounds and a more varied composition with the presence of neochlorogenic acid (19.88 mg L⁻¹) and myricetin (92.87 mg L⁻¹) (Table 2). Homemade traditional *P. pyraeaster* vinegar also had a more complex phenolic composition (gallic acid; neochlorogenic acid; chlorogenic acid; vanillic acid; caffeic acid; epicatechin; *p*-coumaric acid; ferulic acid; sinapic acid; salicylic acid; ellagic acid; rutin; myricetin) compared with commercial vinegars (Tables 2 and 3).

Thanks to its complex composition and higher polyphenol content, the homemade traditional *R. canina* vinegar stands out, compared with the commercial vinegar of the same species (rosehips BIO) or other species (sour cherry, quince, apples Greece, grapes Greece, red wine, and white wine) (Tables 2 and 3). Kalembe-Drożdż et al. [32] mentioned catechin (12.04 mg/100 mL) as being the dominant polyphenol of rosehip vinegar, which is in accordance with the results obtained in this paper regarding the homemade *R. canina* vinegar (674.83 mg L⁻¹). The same authors mentioned chlorogenic acid (0.43 mg/100 mL), ellagic acid (0.62 mg/100 mL), and gallic acid (1.30 mg/100 mL) in rosehip vinegar. As for the homemade vinegars obtained from the genus *Crataegus* (*C. monogyna* and *C. pentag-*

yna), it was found that they are much richer in phenolic compounds compared with the commercial ones (Tables 2 and 3). Compared with the literature, Özdemir et al. [25] confirmed the presence of phenolic compounds in hawthorn vinegar (gallic acid 763.89 mg L^{-1} , chlorogenic acid 534.83 mg L^{-1} , caffeic acid 372.37 mg L^{-1} , catechin 45.65 mg L^{-1} , and epicatechin 39.37 mg L^{-1}). Karadag et al. [34] identified gallic acid (8.55 mg/mL), caffeic acid (12.28 mg/mL), syringic acid (1.71 mg/mL), and rutin (14.7 mg/mL) in hawthorn vinegar.

Regarding the results obtained for commercial vinegars, compared with the literature, the values are different. Grape vinegar is one of the most widespread products in Romania, along with those obtained from white or red wine. Differences between phenolic compounds and their quantity were highlighted between these commercial products (Table 3). For grape vinegar, Kelebek et al. [39] confirmed the presence of gallic acid ($7.45\text{--}21.84 \text{ mg L}^{-1}$), chlorogenic acid ($0.09\text{--}1.77 \text{ mg L}^{-1}$), and vanillic acid ($0\text{--}2.58 \text{ mg L}^{-1}$), values according to those obtained in this study in terms of chlorogenic acid with 0.99 mg L^{-1} and vanillic acid with 0.54 mg L^{-1} , but a lower value of gallic acid (0.25 mg L^{-1}). Jeong et al. [43] identified the presence of gallic acid (0.74 mg/100 mL), chlorogenic acid (0.20 mg/100 mL), catechin (0.78 mg/100 mL), and epicatechin (0.82 mg/100 mL) in Korean grape vinegar. In this paper, the presence of gallic acid (0.25 mg L^{-1}), neochlorogenic acid (3.08 mg L^{-1}), chlorogenic acid (0.99 mg L^{-1}), and vanillic acid (0.54 mg L^{-1}) was highlighted. Red wine vinegar (traditional and industrial) analyzed by Budak and Guzel-Seydim [35] in Turkey confirmed the presence of phenolic compounds such as gallic acid ($16.36\text{--}18.23 \text{ mg L}^{-1}$), catechin ($13.76\text{--}27.50 \text{ mg L}^{-1}$), caffeic acid ($6.30\text{--}10.30 \text{ mg L}^{-1}$), epicatechin ($4.96\text{--}8.20 \text{ mg L}^{-1}$), chlorogenic acid ($3.73\text{--}0.16 \text{ mg L}^{-1}$), syringic acid ($0.70\text{--}0.33 \text{ mg L}^{-1}$), *p*-coumaric acid ($0.23\text{--}0.56 \text{ mg L}^{-1}$), and ferulic acid ($0.06\text{--}0.35 \text{ mg L}^{-1}$), a higher number of compounds phenolics compared with those identified in this study (only gallic acid, catechin, caffeic acid, and *p*-coumaric acid were identified). Liu et al. [42] mentioned, in Chinese red wine vinegar, the presence of gallic acid ($4.10\text{--}9.99 \text{ }\mu\text{g/mL}$), caffeic acid ($1.48\text{--}1.73 \text{ }\mu\text{g/mL}$), and *p*-coumaric acid ($0.81\text{--}1.39 \text{ }\mu\text{g/mL}$). Moreover, *p*-coumaric acid ($0\text{--}0.18 \text{ }\mu\text{g/mL}$), caffeic acid ($0\text{--}0.32 \text{ }\mu\text{g/mL}$), and ferulic acid ($0\text{--}0.31 \text{ }\mu\text{g/mL}$) were identified by Liu et al. [42] in white wine vinegar from China.

In the case of other commercial vinegars obtained from fruits (BIO rosehips, sour cherry, quince, and Greek apples), the results obtained in this study are different from those in the literature. For example, Pashazadeh et al. [31] confirmed the presence of gallic, coumaric, ellagic, catechin, and epicatechin in black rosehip vinegar, and states that the fermentation process is the cause of the decrease in phenol values. Karadag et al. [34] mentioned the presence of gallic acid (8.48 mg/mL) and caffeic acid (12.46 mg/mL) in the studied rosehip vinegar. Regarding sour cherry vinegar, Özen et al. [44] confirmed the presence of gallic acid ($160\text{--}170 \text{ mg/mL}$) and catechin ($0.7\text{--}1 \text{ mg/mL}$) along with chlorogenic acid, *p*-coumaric acid, caffeic acid, ferulic acid, and epicatechin. In the present study, in commercial sour cherry vinegar, only gallic acid (10.73 mg L^{-1}), catechin (1.97 mg L^{-1}), and neochlorogenic acid (1.08 mg L^{-1}) were identified. It turns out that the results on the phenolic composition of vinegars depend on the species and analyzed genotypes, geographical location, timing of fruit harvesting, methods of processing, and analysis.

Owing to the complex composition of vinegars, they can be used with a dual role—food and therapeutic. Homemade wild fruits vinegar, thanks to their content of bioactive compounds, could become functional foods rich in phytochemicals that possess antimicrobial and antioxidant activity with therapeutic effects that could improve or help treat various diseases.

5. Conclusions

The data obtained highlight the high quality of homemade vinegars compared with commercial ones. Various compositions in phenolic compounds and the physical and chemical characteristics cause traditional vinegars obtained from wild fruits, through simple recipes, to be recommended for a healthy diet. Wild fruits used as raw material

can add value to the food sector. The influence of passing time on physical-chemical characteristics of homemade vinegars will be investigated in future studies.

Author Contributions: Conceptualization, S.C. and A.-M.S.; methodology, I.T.; validation, I.T.; investigation, S.C., F.T. and A.-M.S.; data curation, S.C. and A.-M.S.; writing—original draft preparation, S.C., F.T. and A.-M.S.; writing—review and editing, S.C. All authors have read and agreed to the published version of the manuscript.

Funding: This research received no external funding.

Institutional Review Board Statement: Not applicable.

Informed Consent Statement: Not applicable.

Data Availability Statement: Not applicable.

Conflicts of Interest: The authors declare no conflict of interest.

References

- Ousaaïd, D.; Imtara, H.; Laaroussi, H.; Lyoussi, B.; Elarabi, I. An investigation of moroccan vinegars: Their physicochemical properties and antioxidant and antibacterial activities. *J. Food Qual.* **2021**, *2021*, 6618444. [\[CrossRef\]](#)
- Cosmulescu, S.; Trandafir, I.; Nour, V. Phenolic acids and flavonoids profiles of extracts from edible wild fruits and their antioxidant properties. *Int. J. Food Prop.* **2017**, *20*, 3124–3134. [\[CrossRef\]](#)
- Cosmulescu, S.N.; Trandafir, I.; Cornescu, F. Antioxidant capacity, total phenols, total flavonoids and colour component of cornelian cherry (*Cornus mas* L.) Wild genotypes. *Not. Bot. Horti Agrobot.* **2018**, *47*, 390–394. [\[CrossRef\]](#)
- Li, Y.; Zhang, J.-J.; Xu, D.-P.; Zhou, T.; Zhou, Y.; Li, S.; Li, H.-B. Bioactivities and health benefits of wild fruits. *Int. J. Mol. Sci.* **2016**, *17*, 1258. [\[CrossRef\]](#)
- Joshi, G.; Negi, G.C. Quantification and valuation of forest ecosystem services in the western Himalayan region of India. *Int. J. Biodivers. Sci. Ecosyst. Serv. Manag.* **2011**, *7*, 2–11. [\[CrossRef\]](#)
- Helkar, P.B.; Sahoo, A. Review: Food industry by-products used as a functional food ingredients. *Int. J. Waste Resour.* **2016**, *6*, 1–6.
- Cosmulescu, S.; Trandafir, I.; Nour, V.; Ionica, M.; Tutulescu, F. Phenolics content, antioxidant activity and color of green walnut extracts for preparing walnut liquor. *Not. Bot. Horti Agrobot.* **2014**, *42*, 551–555. [\[CrossRef\]](#)
- Bastante, M.J.C.; Guerrero, E.D.; Mejias, R.C.; Marín, R.N.; Dodero, M.C.R.; Barroso, C.G. Study of the polyphenolic composition and antioxidant activity of new sherry vinegar-derived products by maceration with fruits. *J. Agric. Food Chem.* **2010**, *58*, 11814–11820. [\[CrossRef\]](#)
- Zhai, X.; Wang, X.; Wang, X.; Zhang, H.; Ji, Y.; Ren, D.; Lu, J. An efficient method using ultrasound to accelerate aging in crabapple (*Malus asiatica*) vinegar produced from fresh fruit and its influencing mechanism investigation. *Ultrason. Sonochem.* **2021**, *72*, 105464. [\[CrossRef\]](#)
- Samad, A.; Azlan, A.; Ismail, A. Therapeutic effects of vinegar: A review. *Curr. Opin. Food Sci.* **2016**, *8*, 56–61. [\[CrossRef\]](#)
- Budak, N.H.; Aykin, E.; Seydim, A.C.; Greene, A.K.; Güzel-Seydim, Z.B. Functional properties of vinegar. *J. Food Sci.* **2014**, *79*, R757–R764. [\[CrossRef\]](#) [\[PubMed\]](#)
- Ali, Z.; Ma, H.; Rashid, M.T.; Wali, A.; Younas, S. Preliminary study to evaluate the phytochemicals and physiochemical properties in red and black date's vinegar. *Food Sci. Nutr.* **2019**, *7*, 1976–1985. [\[CrossRef\]](#) [\[PubMed\]](#)
- Pinto, T.M.S.; Neves, A.C.C.; Leão, M.V.P.; Jorge, A.O.C. Vinegar as an antimicrobial agent for control of *Candida* spp. in complete denture wearers. *J. Appl. Oral Sci.* **2008**, *16*, 385–390. [\[CrossRef\]](#) [\[PubMed\]](#)
- Yagnik, D.; Serafin, V.; Shah, A.J. Antimicrobial activity of apple cider vinegar against *Escherichia coli*, *Staphylococcus aureus* and *Candida albicans*; downregulating cytokine and microbial protein expression. *Sci. Rep.* **2018**, *8*, 1732. [\[CrossRef\]](#)
- Krusong, W.; Teerarak, M.; Laosinwattana, C. Liquid and vapor-phase vinegar reduces *Klebsiella pneumoniae* on fresh coriander. *Food Control* **2015**, *50*, 502–508. [\[CrossRef\]](#)
- Baldas, B.; Altuner, E.M. The antimicrobial activity of apple cider vinegar and grape vinegar, which are used as a traditional surface disinfectant for fruits and vegetables. *Commun. Fac. Sci. Univ. Ank. Series C* **2018**, *27*, 1–10.
- Chen, H.; Chen, T.; Giudici, P.; Chen, F. Vinegar functions on health: Constituents, sources, and formation mechanisms. *Compr. Rev. Food Sci. Food Saf.* **2016**, *15*, 1124–1138. [\[CrossRef\]](#)
- Raspor, P.; Goranovič, D. Biotechnological applications of acetic acid bacteria. *Crit. Rev. Biotechnol.* **2008**, *28*, 101–124. [\[CrossRef\]](#)
- Amirul, M.S.; Endut, R.; Rashidi, C.B.M.; Aljunid, S.A.; Ali, N.; Laili, M.H.; Laili, A.R.; Ismail, M.N.M. Estimation of harumanis (*Mangifera indica* L.) Sweetness using Near-Infrared (NIR) spectroscopy. *IOP Conf. Series Mater. Sci. Eng.* **2020**, *767*, 012070. [\[CrossRef\]](#)
- Nour, V.; Trandafir, I.; Ionica, M.E. Compositional characteristics of fruits of several apple (*Malus domestica* Borkh.) cultivars. *Not. Bot. Horti Agrobot. Cluj Napoca* **2010**, *38*, 228–233.
- Stoenescu, A.-M.; Trandafir, I.; Cosmulescu, S. Determination of phenolic compounds using HPLC-UV method in wild fruit species. *Horticulturae* **2022**, *8*, 84. [\[CrossRef\]](#)

22. Chang, R.-C.; Lee, H.-C.; Ou, S.-M. Investigation of the physicochemical properties of concentrated fruit vinegar. *J. Food Drug Anal.* **2005**, *13*, 1. [\[CrossRef\]](#)
23. Özdemir, N.; Pashazadeh, H.; Zannou, O.; Koca, I. Phytochemical content, and antioxidant activity, and volatile compounds associated with the aromatic property, of the vinegar produced from rosehip fruit (*Rosa canina* L.). *LWT* **2022**, *154*, 112716. [\[CrossRef\]](#)
24. De la Rosa, L.A.; Moreno-Escamilla, J.O.; Rodrigo-García, J.; Alvarez-Parrilla, E. Phenolic compounds. In *Postharvest Physiology and Biochemistry of Fruits and Vegetables*; Woodhead Publishing: Sawston, UK, 2019; pp. 253–271.
25. Özdemir, G.B.; Özdemir, N.; Ertekin-Filiz, B.; Gökırmaklı, Ç.; Kök-Taş, T.; Budak, N.H. Volatile aroma compounds and bioactive compounds of hawthorn vinegar produced from hawthorn fruit (*Crataegus tanacetifolia* (Lam.) Pers.). *J. Food Biochem.* **2021**, e13676. [\[CrossRef\]](#) [\[PubMed\]](#)
26. Dabija, A.; Hatnean, C.A. Study concerning the quality of apple vinegar obtained through classical method. *J. Agroaliment. Processes Technol.* **2014**, *20*, 304–310.
27. Ousaaïd, D.; Mechchate, H.; Laaroussi, H.; Hano, C.; Bakour, M.; El Ghouizi, A.; Conte, R.; Lyoussi, B.; El Arabi, I. Fruits vinegar: Quality characteristics, phytochemistry, and functionality. *Molecules* **2022**, *27*, 222. [\[CrossRef\]](#)
28. Ozturk, I.; Caliskan, O.; Tornuk, F.; Ozcan, N.; Yalcin, H.; Baslar, M.; Sagdic, O. Antioxidant, antimicrobial, mineral, volatile, physicochemical and microbiological characteristics of traditional home-made Turkish vinegars. *LWT* **2015**, *63*, 144–151. [\[CrossRef\]](#)
29. Matloob, M. Zahdi date vinegar: Production and characterization. *Am. J. Food Technol.* **2014**, *9*, 231–245. [\[CrossRef\]](#)
30. Sung, N.-H.; Woo, S.-M.; Kwon, J.-H.; Yeo, S.-H.; Jeong, Y.-J. Quality characteristics of high acidity apple vinegar manufactured using two stage fermentation. *J. Korean Soc. Food Sci. Nutr.* **2014**, *43*, 877–883. [\[CrossRef\]](#)
31. Pashazadeh, H.; Özdemir, N.; Zannou, O.; Koca, I. Antioxidant capacity, phytochemical compounds, and volatile compounds related to aromatic property of vinegar produced from black rosehip (*Rosa pimpinellifolia* L.) juice. *Food Biosci.* **2021**, *44*, 101318. [\[CrossRef\]](#)
32. Kalembe-Drożdż, M.; Kwiecień, I.; Szewczyk, A.; Cierniak, A.; Grzywacz-Kisiełewska, A. Fermented vinegars from apple peels, raspberries, rosehips, lavender, mint, and rose petals: The composition, antioxidant power, and genoprotective abilities in comparison to acetic macerates, decoctions, and tinctures. *Antioxidants* **2020**, *9*, 1121. [\[CrossRef\]](#) [\[PubMed\]](#)
33. Chochevska, M.; Seniceva, E.J.; Veličkovska, S.K.; Naumova-Letja, G.; Mirčeski, V.; Rocha, J.M.F.; Esatbeyoglu, T. Electrochemical determination of antioxidant capacity of traditional homemade fruit vinegars produced with double spontaneous fermentation. *Microorganisms* **2021**, *9*, 1946. [\[CrossRef\]](#) [\[PubMed\]](#)
34. Karadag, A.; Bozkurt, F.; Bekiroglu, H.; Sagdic, O. Use of principal component analysis and cluster Aanalysis for differentiation of traditionally-manufactured vinegars based on phenolic and volatile profiles, and antioxidant activity. *Polish J. Food Nutr. Sci.* **2020**, *70*, 347–360. [\[CrossRef\]](#)
35. Budak, H.N.; Güzel-Seydim, Z.B. Antioxidant activity and phenolic content of wine vinegars produced by two different techniques. *J. Sci. Food Agric.* **2010**, *90*, 2021–2026. [\[CrossRef\]](#) [\[PubMed\]](#)
36. Beceanu, D.; Anghel, R.M. Assortment of vinegar of supermarkets in Iași: Qualitative assessment according to EU standards. *Cercet. agron. Mold.* **2010**, *43*, 55–64.
37. Kara, M.; Assouguem, A.; El Fadili, M.; Benmessaoud, S.; Alshawwa, S.Z.; Al Kamaly, O.; Saghrouchni, H.; Zerhouni, A.R.; Bahhou, J. Contribution to the evaluation of physicochemical properties, total phenolic content, antioxidant potential, and antimicrobial activity of vinegar commercialized in Morocco. *Molecules* **2022**, *27*, 770. [\[CrossRef\]](#)
38. Ousaaïd, D.; Laaroussi, H.; Mechchate, H.; Bakour, M.; El Ghouizi, A.; Mothana, R.A.; Noman, O.; Es-Safi, I.; Lyoussi, B.; El Arabi, I. The nutritional and antioxidant potential of artisanal and industrial apple vinegars and their ability to inhibit key enzymes related to type 2 diabetes in vitro. *Molecules* **2022**, *27*, 567. [\[CrossRef\]](#)
39. Kelebek, H.; Kadiroğlu, P.; Demircan, N.B.; Selli, S. Screening of bioactive components in grape and apple vinegars: Antioxidant and antimicrobial potential. *J. Inst. Brew.* **2017**, *123*, 407–416. [\[CrossRef\]](#)
40. Nakamura, K.; Ogasawara, Y.; Endou, K.; Fujimori, S.; Koyama, M.; Akano, H. Phenolic compounds responsible for the superoxide dismutase-like activity in high-brix apple vinegar. *J. Agric. Food Chem.* **2010**, *58*, 10124–10132. [\[CrossRef\]](#)
41. Bortolini, D.G.; Benvenuti, L.; Demiate, I.M.; Nogueira, A.; Alberti, A.; Zielinski, A.A.F. A new approach to the use of apple pomace in cider making for the recovery of phenolic compounds. *LWT* **2020**, *126*, 109316. [\[CrossRef\]](#)
42. Liu, Q.; Tang, G.-Y.; Zhao, C.-N.; Gan, R.-Y.; Li, H.-B. Antioxidant activities, phenolic profiles, and organic acid contents of fruit vinegars. *Antioxidants* **2019**, *8*, 78. [\[CrossRef\]](#) [\[PubMed\]](#)
43. Jeong, C.H.; Choi, G.N.; Kim, J.H.; Kwak, J.H.; Kang, S.T.; Choi, S.G.; Heo, H.J. In vitro antioxidant properties and phenolic composition of Korean commercial vinegars. *Food Sci. Biotechnol.* **2009**, *18*, 1258–1262.
44. Özen, M.; Özdemir, N.; Filiz, B.E.; Budak, N.H.; Kök-Taş, T. Sour cherry (*Prunus cerasus* L.) vinegars produced from fresh fruit or juice concentrate: Bioactive compounds, volatile aroma compounds and antioxidant capacities. *Food Chem.* **2020**, *309*, 125664. [\[CrossRef\]](#) [\[PubMed\]](#)